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JAPANESE INDUSTRIAL STANDARD

Iron(III) Oxide for Ferrite

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Iron (III) Oxide for Ferrite

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1. Scope

This Japanese Industrial Standard specifies the iron (III) oxide for ferrite.

Remark: The units and numerical values given in { } in this standard are in accordance with the International System of Units (SI), and are appended for reference.

2. Classification

The iron (III) oxide for ferrite shall be classified into the following three classes according to the contents of main components and other components.

(1) Class 1

(2) Class 2

(3) Class 3

3. Quality

The iron (III) oxide for ferrite shall be tested in accordance with 5. and shall conform to the requirements given in following Table.

Table

Classification Components	Class 1	Class 2	Class 3
Iron (III) oxide (Fe_2O_3) %	99.0 min.	98.8 min.	98.5 min.
Loss on drying %	0.30 max.	0.30 max.	0.30 max.
Silicon dioxide (SiO_2) %	0.01 max.	0.06 max.	0.30 max.
Aluminium (Al) %	0.02 max.	0.02 max.	0.05 max.
Manganese (Mn) %	0.30 max.	0.30 max.	0.30 max.
Calcium (Ca) %	0.01 max.	0.02 max.	0.04 max.
Sulfate ion (SO_4^{2-}) %	—	0.20 max.	0.30 max.
Chloride ion (Cl^-) %	—	0.15 max.	0.15 max.

Applicable Standards: See pages 38 and 39.

4. Sampling Method

The sample shall be so taken as to represent the whole by a reasonable method based on the agreements between the delivery parties concerned.

5. Test Methods

5.1 General Matters General matters shall be as follows:

- (1) General matters common to the chemical analysis shall be in accordance with JIS K 0050.
- (2) General matters common to the absorptiometry shall be in accordance with JIS K 0115.
- (3) General matters common to the atomic absorption spectrophotometry shall be in accordance with JIS K 0121.
- (4) General matters common to the X-ray fluorescence analysis method shall be in accordance with JIS K 0119.

5.2 Iron (III) Oxide

5.2.1 Classification of Determination Methods The methods for determination of iron (III) oxide are classified into the following two methods and either of them may be used.

- (1) Potassium dichromate titrimetric method
- (2) EDTA titrimetric method

5.2.2 Potassium Dichromate Titrimetric Method The potassium dichromate titrimetric method shall be as follows:

- (1) Summary A sample is dissolved with hydrochloric acid and iron (III) ion is reduced for iron (II) ion by adding tin (II) chloride solution. The precipitate of mercury (I) chloride is prepared by further adding mercury (II) chloride solution. Then, the sample solution is added with sodium 4-anilrobenzene sulfonate solution as indicator and titrated with potassium dichromate standard solution.
- (2) Reagents The reagents shall be as follows:
 - (2.1) Hydrochloric Acid The hydrochloric acid specified in JIS K 8180.
 - (2.2) Mixed Acid (Sulfuric Acid - Phosphoric Acid) 140 ml of water is added with 30 ml of sulfuric acid specified in JIS K 8951 while cooling it and further added with 30 ml of phosphoric acid specified in JIS K 9005.
 - (2.3) Tin (II) Chloride Solution 100 ml of hydrochloric acid specified in JIS K 8180 is put into an Erlenmeyer flask, added little by little with 50 g of tin (II) chloride specified in JIS K 8136 while heating it on a water bath, dissolved and diluted

to 1 l by adding water. This solution is added with a small amount of granular tin specified in JIS K 8580, put into a brown bottle, sealed and stored.

- (2.4) Mercury (II) Chloride Solution The saturated solution of mercury (II) chloride specified in JIS K 8139.
- (2.5) Sodium 4-Anilirobenzene Sulfonate Solution (0.2 w/v %) 0.2 g of sodium 4-anilirobenzene sulfonate specified in JIS K 9514 is dissolved with water and diluted to 100 ml. This solution is put into a brown bottle, sealed and stored.
- (2.6) N/10 Potassium Dichromate Standard Solution Potassium dichromate ⁽¹⁾ specified in JIS K 8005 is dried in the oven with thermostat kept at 100 to 110°C for 3 to 4 h and let to stand to cool in a desiccator. 4.903 g thereof (for 100% of $K_2Cr_2O_7$) is weighed out, dissolved with water, transferred into a measuring flask of 1000 ml and added with water up to the marked line.

Note (1) When it is necessary to grind potassium dichromate, an agate mortar is used.

- (3) Operation The operation shall be carried out in accordance with the following procedures:

- (3.1) Weigh out about 4 g of the sample to the nearest 0.1 mg and transfer it into a conical beaker of 300 ml.
- (3.2) Add 40 ml of hydrochloric acid. After dissolving the sample by heating, add 100 ml of water.
- (3.3) After cooling, transfer it into a measuring flask of 250 ml and dilute it up to the marked line with water.
- (3.4) Separately take 20 ml therefrom into a conical beaker of 500 ml and concentrate it with avoiding boiling until the amount of solution becomes about 5 ml.
- (3.5) Drop tin (II) chloride solution until the colour of solution disappears with successively heating it, further add one drop thereof in excess and quickly cool it.
- (3.6) Add 10 ml of mercury (II) chloride solution at one time. After the silky precipitate of mercury (I) chloride is generated, let it stand for about 5 min.
- (3.7) After adding 30 ml of mixed acid (sulfuric acid - phosphoric acid), add 30 to 100 ml of water.
- (3.8) Add 5 to 6 drops of sodium 4-anilirobenzene sulfonate solution (0.02 w/v %) as indicator and immediately titrate it with N/10 potassium dichromate standard solution. When the colour of solution tints purple via from green to blue green and immediately disappears, mix them by shaking each drop and allow the point where the colour of solution does not disappear even if 1/2 to 1/3 drop is added, to be the end point.

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- (3.9) Separately take 3 ml of hydrochloric acid into a conical beaker of 500 ml, dilute it to 25 ml with water, heat it, add one drop of tin (II) chloride solution and quickly cool it. Hereinafter carry out the operation in the same way as that in (3.6) to (3.8) and carry out a blank test.
- (4) Calculation The content of iron (III) oxide (Fe_2O_3) shall be calculated from the following formula:

$$C = \frac{0.007985 \times (V - V')}{S \times \frac{20}{250}} \times 100$$

where

C : iron (III) oxide (%)

0.007985 : amount of iron (III) oxide equivalent to 1 ml of N/10 potassium dichromate standard solution (g)

V : amount of N/10 potassium dichromate standard solution required for titration (ml)

V' : amount of N/10 potassium dichromate standard solution required for blank test (ml)

S : sample (g)

5.2.3 EDTA Titrimetric Method The EDTA titrimetric method shall be as follows:

- (1) Summary A sample is dissolved with hydrochloric acid and nitric acid and its pH is regulated with sodium acetate and aqueous ammonia. Thereafter, it is added with Variamine Blue B as indicator and titrated with EDTA standard solution.
- (2) Reagents The reagents shall be as follows:
- (2.1) Hydrochloric Acid The hydrochloric acid specified in JIS K 8180.
- (2.2) Nitric Acid The nitric acid specified in JIS K 8541.
- (2.3) Hydrochloric Acid (1+3) It is prepared by using hydrochloric acid specified in JIS K 8180.
- (2.4) Aqueous Ammonia (1+2) It is prepared by using aqueous ammonia specified in JIS K 8085.
- (2.5) Sodium Acetate Solution (20 w/v %) 20 g of sodium acetate specified in JIS K 8372 is dissolved with water and diluted to 100 ml.
- (2.6) Variamine Blue B Indicator 1 g of Variamine Blue B hydrochloride [N-(*p*-methoxyphenyl)-*p*-phenylenediamine hydrochloride] and 100 g of sodium chloride specified in JIS K 8150 are mixed until they become homogeneous.

- (2.7) 3/100 M EDTA Standard Solution 11.25 g of disodium ethylene-diaminetetraacetate specified in JIS K 8107 is dissolved with water and whole the amount is diluted to 1 l.

Standardization About 1.5 g of iron (not less than 99.9 %) is weighed out to the nearest 0.1 mg and put into a beaker of 300 ml. It is covered with a watch glass, added with 30 ml of hydrochloric acid specified in JIS K 8180 and 15 ml of nitric acid specified in JIS K 8541 and dissolved. Thereafter, it is transferred into a measuring flask of 500 ml and diluted up to the marked line by adding water. 25 ml therefrom is separately taken into a conical beaker of 500 ml and the operation of (3)(3.4) to (3)(3.7) is carried out. The factor of 3/100 M EDTA standard solution shall be calculated from the following formula:

$$f = \frac{m \times \frac{25}{500}}{V \times 0.001675}$$

where

f : factor of 3/100 M EDTA standard solution

m : weighing out amount of iron (g)

V : amount of 3/100 M EDTA standard solution required for titration (ml)

0.001675: amount of iron equivalent to 1 ml of 3/100 M EDTA standard solution (g)

- (3) Operation The operation shall be carried out in accordance with the following procedures:

- (3.1) Weigh out about 2 g of the sample to the nearest 0.1 mg and transfer it into a conical beaker of 500 ml.
- (3.2) Add 30 ml of hydrochloric acid and 15 ml of nitric acid. After dissolving it by heating, add 200 ml of water.
- (3.3) After cooling, put it into a measuring flask of 500 ml and dilute it up to the marked line by adding water.
- (3.4) Separately take 25 ml therefrom into a conical beaker of 500 ml.
- (3.5) Gradually add aqueous ammonia (1+2). After the precipitate of iron (III) hydroxide is slightly generated, dissolve the precipitate by adding hydrochloric acid (1+3) and make it slightly alkaline. Then, gradually add sodium acetate solution (20 w/v %). After the solution becomes red brown, further add 2 ml.
- (3.6) Drop hydrochloric acid (1+3) until the colour of red brown disappears (2).

- (3.7) Dilute the amount of solution to about 300 to 400 ml by adding warm water, add about 0.05 g of Variamine Blue B indicator and titrate it with 3/100 M EDTA standard solution. Sufficiently mix them by shaking each drop just before the blue purple disappears after the colour of solution becomes gradually pale from blue purple and allow the point where the colour of solution turns yellow via brown by 1/2 to 1/3 drop to be the end point.

Note (2) If operated by this method, its pH will be within a range of 1.7 to 2.0.

- (4) Calculation The content of iron (III) oxide (Fe_2O_3) shall be calculated from the following formula:

$$C = \frac{0.002395 \times f \times V}{S \times \frac{25}{500}} \times 100$$

where

C : iron (III) oxide (%)

0.002395: amount of iron (III) oxide equivalent to 1 ml of 3/100 M EDTA standard solution (g)

f : factor of 3/100 M EDTA standard solution

V : amount of 3/100 M EDTA standard solution required for titration (ml)

S : sample (g)

5.3 Loss on Drying The method for determination of loss on drying shall be as follows:

- (1) Summary A sample is dried in an oven and the loss on drying is obtained.
- (2) Apparatus The apparatus shall be as follows:
- (2.1) Flat Type Weighing Bottle The flat type weighing bottle of 50 mm x 30 mm specified in JIS R 3503.
- (2.2) Oven with Thermostat
- (3) Operation The operation shall be carried out in accordance with the following procedures:
- (3.1) Take about 3 g of the sample into the flat type weighing bottle having a known mass, spread it on the bottom surface of weighing bottle in a fixed thickness as far as possible. After stoppering it, weigh it to the nearest 0.1 mg.
- (3.2) Remove the stopper and dry the weighing bottle and the stopper in the oven with thermostat kept at 105 to 110°C for 2 h.

(3.3) After standing to cool in a desiccator, stopper the bottle. Thereafter, weigh them to the nearest 0.1 mg.

(4) Calculation The loss on drying shall be calculated from the following formula:

$$C = \frac{m_1 - m_2}{S} \times 100$$

where

C: loss on drying (%)

m_1 : mass weighed in (3)(3.1) (g)

m_2 : mass weighed in (3)(3.3) (g)

S: sample (g)

5.4 Silicon Dioxide

5.4.1 Classification of Determination Methods The methods for determination of silicon dioxide are classified into the following two methods and either of them may be used.

(1) Absorptiometry

(2) Gravimetric Method

5.4.2 Absorptiometry The absorptiometry shall be as follows:

(1) Summary After dissolving a sample with hydrochloric acid, it is added with perchloric acid, oxidized and dehydrated. After filtering, the precipitate is fused with sodium carbonate. This solution is added with ammonium molybdate solution and made to be dodecamolybdosilicate. It is added with reducing agent and the absorbance of the generated molybdenum blue is measured. Then, silicon dioxide is obtained from the working curve.

(2) Reagents The reagents shall be as follows:

(2.1) Hydrochloric Acid The hydrochloric acid specified in JIS K 8180.

(2.2) Hydrochloric Acid (1+1) It is prepared by using hydrochloric acid specified in JIS K 8180.

(2.3) Hydrochloric Acid (1+10) It is prepared by using hydrochloric acid specified in JIS K 8180.

(2.4) Perchloric Acid The perchloric acid of 60 % in content specified in JIS K 8223.

(2.5) Ammonium Molybdate Solution (10 w/v %) 10 g of ammonium molybdate specified in JIS K 8905 is dissolved with water and diluted to 100 ml.

(2.6) Sodium Carbonate The sodium carbonate specified in JIS K 8625.

- (2.7) Tartaric Acid Solution (10 w/v %) 10 g of tartaric acid specified in JIS K 8532 is dissolved with water and diluted to 100 ml.
- (2.8) Sulfuric Acid (1+11) It is prepared by using sulfuric acid specified in JIS K 8951.
- (2.9) Reducing Agent Solution 0.5 g of 1-amino-2-naphthol-4-sulfonic acid specified in JIS K 8050, 2 g of sodium sulfite specified in JIS K 8061 and 20 g of sodium hydrogen sulfite specified in JIS K 8059 are dissolved with 200 ml of water and stored in a cold dark place.
- (2.10) Silicon Dioxide Standard Stock Solution (0.1 mg SiO₂/ml) After igniting silicic acid anhydride specified in JIS K 8885 at 1000°C to constant weight, it stands to cool in a desiccator. 0.10 g is weighed out therefrom, fused in a platinum crucible together with 1 g of sodium carbonate specified in JIS K 8625, dissolved with warm water, transferred into a measuring flask of 1000 ml and cooled. Thereafter, it is diluted up to the marked line by adding water and stored in a polyethylene vessel.
- (2.11) Silicon Dioxide Standard Solution (0.005 mg SiO₂/ml) 10 ml of silicon dioxide standard stock solution (0.1 mg SiO₂/ml) is separately taken into a measuring flask of 200 ml and diluted up to the marked line by adding water. This solution shall be prepared at each use.
- (3) Apparatus and Filter Paper The apparatus and filter paper shall be as follows:
- (3.1) Platinum Crucible The platinum crucible specified in JIS H 6201.
- (3.2) Photoelectric Photometer or Photoelectric Spectrophotometer
- (3.3) Filter Paper The filter paper of Class 6 for quantitative analysis specified in JIS P 3801.
- (4) Operation The operation shall be carried out in accordance with the following procedures:
- (4.1) Take about 10 g of the sample for Class 1, about 3 g for Class 2 and about 1.5 g for Class 3, weigh each of them to the nearest 0.01 g and transfer it into a beaker of 300 ml.
- (4.2) Add 50 ml of hydrochloric acid for Class 1 and 30 ml for Class 2 and Class 3, cover each of them with a watch glass and dissolve it by heating it on a hot plate.
- (4.3) Add 50 ml of perchloric acid for Class 1 and 30 ml for Class 2 and Class 3. Ignite each of them and continue to heat it for about 15 min to the degree that the evaporated vapour of perchloric acid is refluxed along the wall of beaker.

- (4.4) Put down the beaker from the hot plate, let it stand to cool, add 40 ml of hydrochloric acid (1+1) to the deposited perchlorates for Class 1 and 20 ml for Classes 2 and 3, dilute whole the amount to about 200 ml with warm water and completely dissolve it by heating it with agitating.
- (4.5) Filter it with a filter paper, wash the beaker and the filter paper with warm water and warm hydrochloric acid (1+10) until the colour attached to the filter paper disappears and further wash them two to three times with warm water.
- (4.6) Transfer the precipitate together with the filter paper into a platinum crucible and completely incinerate it at about 500°C.
- (4.7) After cooling, weigh out 1 g of sodium carbonate to the nearest 0.01 g, put it into the former platinum crucible and fuse it for about 5 min.
- (4.8) After standing to cool, add 20 ml of water dissolve it by heating on a hot plate and transfer it into a beaker of 300 ml.
- (4.9) Add 10 ml of sulfuric acid (1+11) and agitate them to mix.
- (4.10) After cooling, transfer it into a measuring flask of 250 ml with washing and dilute it up to the marked line with water.
- (4.11) Separately take 5 to 25 ml (so as to contain 0.001 to 0.3 w/v % as SiO_2) therefrom into a measuring flask of 50 ml according to the estimated content of silicon dioxide and dilute it to about 30 ml by adding water.
- (4.12) Add 2 ml of sulfuric acid (1+11) and 2 ml of ammonium molybdate solution (10 w/v %) and sufficiently mix them by shaking. Thereafter, let it stand for 10 min.
- (4.13) Add 4 ml of tartaric acid solution (10 w/v %), mix them by shaking, add 2 ml of reducing agent solution within 1 min, mix them by shaking and add water up to the marked line.
- (4.14) After standing for about 10 min, transfer it into an absorption cell, measure the absorbancy at near 800 nm in wavelength utilizing the solution of blank test as contrast solution and obtain silicon dioxide (SiO_2) from the working curve preliminarily prepared.
- (5) Preparation of Working Curve Stepwise take not less than 5 specimens of silicon dioxide standard solutions (0.005 mg SiO_2 /ml) within a range of 0 to 30 ml thereof into measuring flasks of 50 ml, carry out the operation on and after (4)(4.11), obtain the absorbancy and prepare the working curve indicating the relation between the amount of silicon dioxide and the absorbancy.

5.4.3 Gravimetric Method The gravimetric method shall be as follows:

- (1) **Summary** A sample is dissolved with hydrochloric acid and nitric acid, added with perchloric acid, dehydrated and filtered. Thereafter, it is added with hydrofluoric acid and silicon dioxide is volatilized. Then, its loss is obtained as silicon dioxide.
- (2) **Reagents** The reagents shall be as follows:
 - (2.1) **Hydrochloric Acid** The hydrochloric acid specified in JIS K 8180.
 - (2.2) **Nitric Acid** The nitric acid specified in JIS K 8541.
 - (2.3) **Perchloric Acid** The perchloric acid of 60 % in content specified in JIS K 8223.
 - (2.4) **Hydrochloric Acid (1+1)** It is prepared by using hydrochloric acid specified in JIS K 8180.
 - (2.5) **Hydrochloric Acid (1+10)** It is prepared by using hydrochloric acid specified in JIS K 8180.
 - (2.6) **Sulfuric acid (1+1)** It is prepared by using sulfuric acid specified in JIS K 8951.
 - (2.7) **Hydrofluoric Acid** The hydrofluoric acid specified in JIS K 8819.
- (3) **Apparatus and Filter Paper** The apparatus and filter paper shall be as follows:
 - (3.1) **Platinum Crucible** The platinum crucible specified in JIS H 6201.
 - (3.2) **Filter Paper** The filter paper of Class 6 for quantitative analysis specified in JIS P 3801.
- (4) **Operation** The operation shall be carried out in accordance with the following procedures:
 - (4.1) Take about 30 g of the sample for Class 1, about 10 g for Class 2 and about 5 g for Class 3, weigh it to the nearest 0.01 g and transfer it into a wide-mouthed beaker of 500 ml for Class 1 and into a wide-mouthed beaker of 300 ml for Classes 2 and 3.
 - (4.2) Add 50 ml of water, then add 100 ml of hydrochloric acid and 30 ml of nitric acid for Class 1 and 40 ml of hydrochloric acid and 10 ml of nitric acid for Classes 2 and 3, dissolve it by heating on a hot plate, successively heat it and concentrate it to about 150 ml for Class 1 and to about 80 ml for Classes 2 and 3.
 - (4.3) Add 230 ml of perchloric acid for Class 1 and 120 ml for Classes 2 and 3 and ignite them until perchlorates is deposited (³).

- (4.4) Put down the wide-mouthed beaker from the hot plate, let it stand to cool, add 60 ml of hydrochloric acid (1+1) to the deposited perchlorates for Class 1 and 30 ml for Classes 2 and 3, dilute whole the amount to about 300 ml with warm water for Class 1 and to about 200 ml for Classes 2 and 3 and completely dissolve it while mixing it with stirring.
- (4.5) Immediately filter it with a filter paper, rub off the residue attached to the inside wall of beaker with the glass rod with rubber tube, transfer it onto the filter paper, wash the beaker and the filter paper with warm hydrochloric acid (1+10) until the colour adhered to the filter paper disappears and further wash them 4 to 5 times with warm water.
- (4.6) Transfer the precipitate together with the filter paper into a platinum crucible. After drying, incinerate the filter paper at about 500°C, ignite it at 1100°C and over to constant weight (⁴) and let it stand to cool in a desiccator. Thereafter, weigh it to the nearest 0.1 mg.
- (4.7) Wet the residue with 2 to 3 drops of sulfuric acid (1+1), add about 2 ml of hydrofluoric acid and heat it on a hot plate until white fume of sulfuric acid is not generated.
- (4.8) Ignite it again at 1100°C and over to constant weight and let it stand to cool in a desiccator. Thereafter, weigh it to the nearest 0.1 mg.
- (4.9) Separately carry out the operation of (4.2) to (4.8) and calculate the value of blank test from the following formula:

$$B = m_1' - m_2'$$

where

B : value of blank test (g)

m_1' : mass weighed by the operation (4.6) of blank test (g)

m_2' : mass weighed by the operation (4.8) of blank test (g)

Notes (³) The point where perchlorates turn purple when let to stand to cool, shall be made to be the end point.

(⁴) It shall be gently heated until the filter paper burns out and ignited thereafter.

- (5) Calculation The content of silicon dioxide (SiO_2) shall be calculated from the following formula: